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PROCESSING AND EVALUATION OF INTERLABORATORY ANALYTICAL RESULTS FOR THE CERTIFICATION OF ZINC-COPPER ORE RU-1 AS A REFERENCE MATERIAL

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PROCESSING AND EVALUATION OF INTERLABORATORY ANALYTICAL RESULTS FOR THE CERTIFICATION OF ZINC-COPPER ORE RU-1 AS A REFERENCE MATERIAL

by

W.S. Bowman & R. Sutarno

INTRODUCTION

The characterization and certification of zinc-copper ore, RU-1, is a facet of the Ores Task Force of the Canadian Certified Reference Materials Project (CCRMP). An account of the preparation of this reference material and the statistical treatments that were applied to the interlaboratory results is being prepared[1] for distribution with the bottles of the material as they are issued to users.

This report, which is intended to serve as an internal reference for CANMET personnel, gives detailed records of the data processing and statistical treatments which were used for RU-1. Much of this information is not included in the certification document[1].

Participating Laboratories

The names of the contributing laboratories and the lab numbers assigned to them are listed below. Gaps in the list result from the fact that certain laboratories agreed to participate and then found that they could not meet the deadline.

Lab - 1	Sherritt Gordon Mines Limited, Research and Development Division, Fort Saskatchewan, Alberta.
Lab - 2	CANMET #1; Ores and Fire Assay Section, Mineral Sciences Laboratories.
Lab - 3	CANMET #2; Special Analyses Section, Mineral Sciences Laboratories.
Lab - 4	Sherritt Gordon Mines Limited, Mining and Milling Division, Lynn Lake, Manitoba.
Lab - 5	Hudson Bay Mining and Smelting Company Limited, Flin Flon, Manitoba.
Lab - 6	Noranda Mines Limited, Noranda, Quebec.
Lab - 7	Lakefield Research of Canada Limited, Lakefield, Ontario.
Lab - 8	Ministry of Natural Resources, Mineral Research Branch, Toronto, Ontario.
Lab - 10	Thunder Bay Testing Limited, Thunder Bay, Ontario.
Lab - 11	Bondar-Clegg and Company Limited, Ottawa, Ontario.
Lab - 12	Bondar-Clegg and Company Limited, Vancouver, British Columbia.
Lab - 13	British Columbia Department of Mines and Petroleum Resources, Victoria, British Columbia.
Lab - 14	Chemex Labs Limited, North Vancouver, British Columbia.
Lab - 15	Can Test Limited, Vancouver, British Columbia.
Lab - 16	Falconbridge Nickel Mines Limited, Metallurgical Laboratories, Thornhill, Ontario.
Lab - 18	Loring Laboratories Limited, Calgary, Alberta.
Lab - 19	Cominco Limited, Trail, British Columbia.
Lab - 20	LKAB, Kiruna, Sweden.
Lab - 22	Inco Limited, Analytical Services, Process Technology, Copper Cliff, Ontario.
Lab - 24	CANMET #3, Spectrochemistry Section, Mineral Sciences Laboratories.
Lab - 25	Manitoba Department of Mines, Resources and Environmental Management, Mines Branch, Winnipeg, Manitoba.

Lab - 26 Geological Survey of Canada, Central Laboratories and Administrative Services, Ottawa, Ontario.

Lab - 27 Ministère des Richesses Naturelles, Centre de Recherches Minérales, Ste-Foy, Québec.

Lab - 28 CANMET #4; Solution Chemistry Section, Mineral Sciences Laboratories.

Lab - 29 United Keno Hill Mines Limited, Elsa, Yukon.

Data Processing

The analytical results for RU-1 were processed in the same manner as for other CCRMP standards, i.e., by the use of a Dept. of EMR-CDC computer system feature called UPDATE[2,3]. This feature uses card images on either disk or tape. Development of the data set and production runs were done on disk files, while the final copy, which is retained indefinitely, resides in the EMR tape library. The following information is required to access the tape for RU-1:

VSN = ER8306

L = RU1

D = PE (9-track)

UPDATE provides a sequence number for each card image for RU-1. These are grouped by element into four decks: Zn, Cu, Fe, and S. A hard copy of the card images, including sequence numbers, is kept by the authors.* A new copy can be generated, if necessary, from the tape file.

*Copies of this UPDATE Compile File for RU-1 are available from the authors.

Detection of Outliers

All of the results reported by the participating laboratories are listed in Tables 1 (a) to 1 (d) and are presented graphically in Figures 1 (a) to 1 (d). Sets of results whose means differ by more than twice the overall standard deviation from the consensus value and sets whose coefficients of variation were unusually large were considered to be outliers and were excluded from subsequent computations. Also, three sets of combustion sulphur results were arbitrarily excluded. These outliers are noted in Table 1 and circled in Figure 1. The UPDATE correction set for the deletion of these so-called statistical outliers follows along with the list of the sets affected:

```
*IDENT    STAT
*DELETE   ZN.40, Zn.63          Zinc - lab-5(a.a.), lab-5(vol.)
*DELETE   MOD6.61, MOD6.84      Zinc - lab-26(spectr.1),
                                       lab-26(spectr.2)
*DELETE   MOD6.133, MOD6.156    Copper - lab-26(spectr.1),
                                         lab-26(spectr.2)
*DELETE   FE.40, FE.51          Iron - lab-6(vol.)
*DELETE   MOD2.182, MOD2.193    Sulphur - lab-12(comb.)
*DELETE   MOD6.205, MOD6.228    Sulphur - lab-19(grav.), lab-19(comb.)
*DELETE   S.100, MOD4.183       Sulphur - lab-22(comb.)
```

Confirmation of Homogeneity using Interlaboratory Results

Tables 2 (a) to 2 (d) give the means, coefficients of variation, and summary of the t-test between bottles at the 5% significance level for all sets of results for RU-1. The same information is shown graphically in Figures 2 (a) to 2 (d)

in which, for each set, the difference between the means of the two bottles is plotted against the corresponding mean of the results for both bottles. The vertical bar represents the 95% confidence interval of the former. If a bar intersects the abscissa, the null hypothesis is accepted, i.e., there is no evidence of inhomogeneity between bottles for that set of results. In Figure 2, the outliers noted above are not shown except in the case of sulphur (Figure 2 (d)) where sets by the combustion method are indicated by the letter "C". Thus to produce Figures 2 (a) and 2 (d), the UPDATE correction set shown above, minus the last three directives, is used.

Table 2 and Figure 2 show some evidence of between-bottle inhomogeneity. This was confirmed for zinc, copper and iron by using a two-way ANOVA technique with nested design[4]. For the two-way ANOVA, the outliers noted above were excluded. Two copper sets for one bottle only were also excluded:

```
*DELETE MOD4.49, MOD4.62
```

The two-way ANOVA tables and the estimates of between-set, between-bottle, and within-bottle standard deviations (PSI, OMEGA, and SIGMA, respectively) are given in Tables 3 (a) to 3 (d).

Although it is clear that between-bottle differences do exist, they are small compared to the large between-set component of variance. It is considered that RU-1 should be suitably homogeneous for most applications.

Estimation of Consensus Values and Related Statistical Parameters

A one-way ANOVA technique[4] was used to calculate the consensus values and to estimate the variance of the means and

hence the 95% confidence limits. This technique permits the use of the two copper sets referred to above. The mathematical model and general ANOVA table are reported elsewhere[1]. Sets of results identified as outliers on page 4 were not used for these computations. The computed statistics, including the consensus values and confidence limits, are given in Table 4. The certification factors for the four elements are all less then critical value of 4. Thus, the consensus values are accepted as recommended values for RU-1.

REFERENCES

1. FAYE, G.H., BOWMAN, W.S. and SUTARNO, R., Zinc-copper ore, RU-1: Its characterization and preparation for use as a certified reference material. CANMET report CM 77-7.
2. Control Data Cyber 70 UPDATE Reference Manual, Control Data Corporation Publication No.60342500.
3. BOWMAN, W.S., The use of UPDATE to process data files, Mineral Sciences Laboratories Report MRP/MSL 75-176(TR).
4. BROWNLEE, K.A., Statistical Theory and methodology in sciences. John Wiley and Sons, Inc., New York (1961).

Table 1(a)

Zinc results for RU-1

ZINC (WEIGHT PERCENT)

											SAMPLE WT. G
LAB- 1 (A.A.)	2.250	2.250	2.270	2.260	2.260	2.280	2.280	2.280	2.260	2.260	1.0
LAB- 2 (A.A.)	2.210	2.220	2.220	2.240	2.220	2.230	2.230	2.200	2.220	2.230	0.5
LAB- 2 (VOL.)	2.290	2.280	2.290	2.270	2.290	2.270	2.270	2.280	2.290	2.280	0.5
LAB- 3 (POLAR.)	2.210	2.210	2.190	2.190	2.260	2.220	2.220	2.200	2.240	2.240	0.5
LAB- 4 (A.A.)	2.270	2.270	2.270	2.270	2.260	2.270	2.280	2.270	2.280	2.270	2.0
LAB- 4 (VOL.)	2.253	2.270	2.260	2.270	2.270	2.250	2.250	2.270	2.240	2.260	2.0
* LAB- 5 (A.A.)	2.000	2.100	2.000	2.100	2.100	2.000	2.100	2.000	2.100	2.000	0.2
* LAB- 5 (VOL.)	2.000	2.000	2.100	2.000	2.000	2.000	2.000	2.000	2.000	2.000	2.0
LAB- 6 (A.A.)	2.280	2.270	2.300	2.260	2.240	2.280	2.260	2.270	2.280	2.300	0.5
LAB- 6 (POLAR.)	2.320	2.340	2.340	2.320	2.350	2.240	2.240	2.270	2.250	2.270	1.0
LAB- 7 (A.A.)	2.216	2.220	2.230	2.225	2.229	2.220	2.230	2.222	2.230	2.234	1.0
LAB- 8 (A.A.)	2.200	2.210	2.200	2.210	2.210	2.190	2.200	2.200	2.210	2.200	0.5
LAB-10 (A.A.)	2.320	2.270	2.340	2.300	2.360	2.280	2.310	2.330	2.330	2.320	0.5
LAB-10 (VOL.)	2.260	2.310	2.280	2.260	2.250	2.270	2.320	2.300	2.280	2.260	1.0
LAB-11 (A.A.)	2.240	2.230	2.250	2.230	2.250	2.240	2.250	2.240	2.250	2.250	0.5
LAB-12 (A.A.)	2.120	2.150	2.160	2.150	2.190	2.170	2.120	2.160	2.160	2.150	0.5
LAB-12 (VOL.)	2.200	2.210	2.210	2.200	2.120	2.200	2.200	2.220	2.220	2.210	2.0
LAB-13 (A.A.)	2.264	2.274	2.279	2.286	2.287	2.274	2.273	2.284	2.277	2.277	1.0
LAB-14 (A.A.)	2.300	2.273	2.257	2.293	2.267	2.266	2.287	2.273	2.254	2.265	1.0
LAB-15 (A.A.)	2.150	2.150	2.160	2.140	2.140	2.140	2.150	2.150	2.140	2.150	0.5
LAB-15 (VOL.)	2.180	2.160	2.160	2.170	2.170	2.150	2.180	2.170	2.150	2.160	2.0
LAB-16 (A.A.)	2.250	2.270	2.270	2.230	2.230	2.240	2.240	2.200	2.210	2.210	0.25
LAB-18 (A.A.)	2.230	2.230	2.250	2.230	2.230	2.250	2.230	2.230	2.230	2.230	0.5
LAB-19 (A.A.)	2.260	2.200	2.220	2.200	2.200	2.260	2.200	2.220	2.210	2.190	1.0
LAB-19 (VOL.)	2.160	2.150	2.180	2.150	2.170	2.160	2.150	2.170	2.170	2.170	2.0
LAB-20 (A.A.)	2.220	2.260	2.270	2.260	2.260	2.220	2.260	2.230	2.240	2.260	0.2
LAB-22 (A.A.-1)	2.240	2.220	2.240	2.240	2.240	2.240	2.240	2.240	2.230	2.240	0.5
LAB-22 (A.A.-2)	2.220	2.220	2.220	2.210	2.220	2.230	2.230	2.230	2.220	2.220	0.5
LAB-25 (A.A.)	2.220	2.240	2.250	2.240	2.250	2.230	2.260	2.230	2.220	2.240	1.0
* LAB-26 (SPECTR.1)	2.100	2.330	2.310	2.050	2.160	2.250	1.990	1.900	2.040	1.800	0.6
* LAB-26 (SPECTR.2)	1.840	2.160	2.280	1.950	1.890	1.940	2.110	2.360	1.910	1.990	0.1
LAB-27 (A.A.)	2.220	2.240	2.250	2.200	2.200	2.240	2.220	2.220	2.250	2.240	0.25
LAB-28 (A.A.)	2.250	2.250	2.250	2.250	2.250	2.250	2.240	2.250	2.250	2.260	1.0
LAB-29 (A.A.)	2.260	2.260	2.310	2.300	2.270	2.260	2.250	2.300	2.280	2.260	0.2

* Sets judged to be outliers.

Table 1(b)

Copper results for RU-1

COPPER (WEIGHT PERCENT)

											SAMPLE WT. G
LAB- 1 (A.A.)	.873	.876	.884	.882	.881	.883	.878	.880	.869	.881	1.0
LAB- 2 (A.A.-1)	.868	.886	.868	.877	.877	.860	.873	.868	.873	.868	0.5
LAB- 2 (A.A.-2)	.889	.891	.890	.886	.890	.919	.911	.897	.913	.897	0.5
LAB- 2 (COLOR.)	.889	.879	.898	.900	.879	.853	.863	.879	.868	.874	0.5
LAB- 3 (POLAR.)	.838	.847	.842	.833	.857	.848	.843	.845	.852	.846	0.5
LAB- 4 (A.A.)	.856	.860	.856	.854	.855	.855	.860	.853	.855	.851	2.0
LAB- 4 (VOL.)	.850	.850	.850	.860	.860	.850	.860	.860	.860	.860	2.0
LAB- 5 (A.A.)	.820	.830	.820	.820	.830	.810	.810	.800	.800	.800	0.2
LAB- 6 (A.A.)	.830	.820	.840	.790	.850	.830	.820	.830	.790	.840	0.5
LAB- 6 (POLAR.)	.840	.850	.870	.850	.850	.860	.850	.850	.850	.830	1.0
LAB- 7 (A.A.)	.862	.858	.860	.864	.856	.858	.864	.860	.860	.862	1.0
LAB- 8 (A.A.)	.850	.860	.860	.850	.850	.860	.860	.850	.860	.860	0.5
LAB-10 (A.A.)	.860	.840	.820	.840	.870	.820	.890	.880	.830	.850	1.0
LAB-10 (VOL.)	.830	.840	.830	.870	.860	.840	.830	.880	.830	.860	1.0
LAB-11 (A.A.)	.860	.850	.850	.855	.855	.860	.855	.860	.860	.860	0.5
LAB-12 (A.A.-1)	.834	.834	.834	.834	.836	.834	.834	.840	.840	.847	0.5
LAB-12 (A.A.-2)	.870	.854	.870	.872	.870	.850	.866	.862	.858	.860	1.0
LAB-12 (VOL.)	.838	.850	.838	.850	.838	.838	.838	.838	.850	.850	2.0
LAB-13 (A.A.)	.864	.871	.868	.877	.872	.875	.870	.874	.874	.870	1.0
LAB-13 (VOL.)	.865	.870	.858	.882	.868	.878	.868	.868	.871	.876	1.0
LAB-14 (A.A.)	.845	.845	.847	.855	.843						1.0
LAB-14 (COLOR.)	.868	.855	.834	.838	.851						0.5
LAB-15 (A.A.)	.846	.848	.850	.848	.846	.850	.848	.852	.852	.848	0.5
LAB-16 (A.A.)	.876	.860	.860	.840	.845	.856	.852	.856	.855	.860	0.25
LAB-18 (A.A.)	.850	.850	.840	.850	.840	.870	.850	.850	.850	.860	0.5
LAB-19 (A.A.)	.840	.860	.850	.840	.860	.860	.860	.850	.850	.860	1.0
LAB-19 (VOL.)	.870	.870	.870	.870	.870	.870	.870	.870	.870	.880	2.5
LAB-20 (A.A.)	.843	.835	.840	.845	.836	.835	.843	.836	.836	.833	0.2
LAB-22 (A.A.)	.829	.834	.840	.838	.826	.836	.838	.825	.842	.828	0.5
LAB-22 (VOL.)	.814	.834	.834	.834	.834	.834	.834	.827	.834	.820	2.0
LAB-24 (X.R.F.)	.895	.896	.904	.875	.881	.875	.874	.876	.876	.875	**
LAB-25 (A.A.)	.850	.830	.830	.830	.830	.850	.830	.830	.820	.830	1.0
* LAB-26 (SPECTR.1)	.940	.940	.900	.970	.940	.940	.860	.900	.910	.860	0.6
* LAB-26 (SPECTR.2)	.769	.869	.907	.868	.808	.880	.908	.860	.757	.840	0.1
LAB-27 (A.A.)	.860	.850	.880	.860	.880	.890	.860	.870	.880	.890	0.25
LAB-28 (A.A.)	.853	.851	.849	.852	.849	.850	.848	.850	.844	.843	1.0
LAB-29 (A.A.)	.850	.850	.860	.860	.850	.860	.860	.850	.860	.850	0.1

*Sets judged to be outliers.

**5.0 g pellet

Table 1(c)

Iron results for RU-1

	IRON (WEIGHT PERCENT)										SAMPLE WT. G
LAB- 1 (VOL.)	24.60	24.50	24.60	24.60	24.60	24.60	24.60	24.60	24.60	24.60	0.5
LAB- 2 (VOL.)	24.48	24.43	24.43	24.37	24.43	24.48	24.37	24.37	24.48	24.43	0.5
LAB- 3 (VOL.)	24.43	24.44	24.42	24.44	24.44	24.43	24.42	24.43	24.45	24.44	0.5
LAB- 4 (VOL.)	24.43	24.38	24.40	24.35	24.35	24.35	24.38	24.35	24.33	24.35	0.5
LAB- 5 (VOL.)	24.30	24.30	24.30	24.30	24.30	24.30	24.30	24.30	24.30	24.20	0.5
*LAB- 6 (VOL.)	24.00	23.90	24.00	24.00	24.00	23.90	24.00	24.00	24.00	24.00	0.5
LAB- 7 (VOL.)	24.31	24.41	24.30	24.33	24.31	24.42	24.43	24.43	24.40	24.40	0.5
LAB- 8 (VOL.)	24.18	24.13	24.21	24.24	24.18	24.18	24.13	24.27	24.24	24.21	1.0
LAB-10 (VOL.)	24.74	24.72	24.74	24.70	24.60	24.72	24.70	24.59	24.69	24.67	1.0
LAB-11 (VOL.)	24.31	24.30	24.30	24.36	24.33	24.21	24.19	24.28	24.17	24.18	1.0
LAB-12 (VOL.)	24.65	24.75	24.75	24.65	24.65	24.65	24.65	24.65	24.65	24.65	0.25
LAB-13 (A.A.)	24.16	24.04	24.19	24.32	24.22	24.27	24.29	24.40	24.55	24.38	1.0
LAB-14 (VOL.)	24.24	24.16	24.23	24.10	24.11	24.30	24.21	24.18	24.30	24.34	0.5
LAB-15 (VOL.)	24.75	24.75	24.70	24.80	24.75	24.75	24.75	24.70	24.75	24.80	0.5
LAB-16 (VOL.)	24.27	24.31	24.33	24.32	24.35	24.23	24.28	24.36	24.25	24.29	0.4
LAB-18 (VOL.)	24.39	24.44	24.44	24.44	24.39	24.44	24.44	24.39	24.44	24.44	0.5
LAB-19 (VOL.)	24.44	24.44	24.42	24.42	24.44	24.42	24.42	24.42	24.40	24.44	0.5
LAB-20 (VOL.)	24.29	24.30	24.24	24.32	24.29	24.32	24.28	24.30	24.20	24.28	0.5
LAB-22 (VOL.)	24.40	24.55	24.40	24.55	24.50	24.50	24.35	24.55	24.50	24.45	0.5
LAB-22 (A.A.)	24.45	24.35	24.25	24.25	24.35	24.35	24.35	24.25	24.45	24.35	0.5
LAB-25 (VOL.)	24.40	24.50	24.60	24.50	24.40	24.20	24.10	24.20	24.40	24.50	0.4
LAB-27 (A.A.)	24.60	24.60	24.60	24.60	24.60	24.70	24.60	24.80	24.60	24.60	0.25
LAB-27 (VOL.)	24.36	24.22	24.36	24.22	24.22	24.22	24.08	23.94	24.22	24.08	0.5
LAB-27 (VOL.-P.)	24.27	24.27	24.32	24.27	24.38	24.10	24.16	24.13	24.27	24.05	0.5
LAB-29 (VOL.)	24.47	24.42	24.45	24.45	24.47	24.57	24.52	24.57	24.54	24.52	1.0

*Set judged to be outlier.

Table 1(d)

Sulphur results for RU-1

SULPHUR (WEIGHT PERCENT)

											SAMPLE WT. G
LAB- 1 (GRAV.)	21.80	22.10	21.70	22.30	22.20	21.70	21.70	22.20	22.30	22.20	0.5
LAB- 2 (GRAV.)	21.50	21.59	21.53	21.51	21.59	21.51	21.51	21.59	21.49	21.50	0.5
LAB- 3 (GRAV.)	21.56	21.61	21.54	21.54	21.54	21.54	21.51	21.53	21.51	21.55	0.3
LAB- 4 (GRAV.)	21.47	21.49	21.37	21.44	21.46	21.31	21.39	21.47	21.48	21.44	0.5
LAB- 5 (GRAV.)	21.61	21.67	21.64	21.66	21.59	21.67	21.62	21.59	21.67	21.67	0.5
LAB- 6 (GRAV.)	22.15	22.19	22.16	22.14	22.18	22.10	22.13	22.07	22.07	22.09	0.5
LAB- 7 (GRAV.)	21.60	21.54	21.60	21.56	21.48	21.62	21.64	21.56	21.50	21.49	1.0
LAB- 8 (GRAV.)	21.10	21.30	21.21	21.23	21.17	21.18	21.26	21.15	21.14	21.32	0.5
LAB-11 (GRAV.)	21.62	21.56	21.57	21.47	21.70	21.64	21.54	21.67	21.54	21.68	0.4
LAB-12 (GRAV.)	21.93	21.87	21.82	21.83	21.82	21.80	21.78	21.78	21.75	21.86	0.5
*LAB-12 (COMB.)	22.42	22.12	22.39	22.45	22.45	22.20	21.95	22.23	21.69	22.23	0.01
LAB-14 (GRAV.)	21.89	21.64	21.69	21.63	21.61	21.52	21.71	21.77	21.52	21.38	0.2
LAB-16 (GRAV.)	21.29	21.33	21.28	21.35	21.25	21.30	21.28	21.31	21.29	21.37	0.4
LAB-18 (GRAV.)	21.71	21.60	21.59	21.59	21.60	21.72	21.67	21.67	21.54	21.67	0.5
*LAB-19 (GRAV.)	22.30	22.30	22.30	22.10	22.10	22.10	22.30	22.30	22.20	22.20	0.25
*LAB-19 (COMB.)	22.40	22.40	22.20	22.40	22.40	22.40	22.00	22.40	22.40	22.00	0.05
LAB-22 (GRAV.)	21.64	21.42	21.53	21.69	21.64	21.50	21.50	21.58	21.64	21.64	0.5
*LAB-22 (COMB.)	21.60	21.60	21.60	21.70	21.70	21.70	21.70	21.80	21.70	21.60	0.1
LAB-25 (GRAV.)	21.60	21.57	21.68	21.50	21.73	21.66	21.67	21.59	21.59	21.59	0.5
LAB-27 (GRAV.)	21.58	21.61	21.59	21.50	21.54	21.65	21.58	21.68	21.67	21.61	0.5

*Sets judged to be outliers.

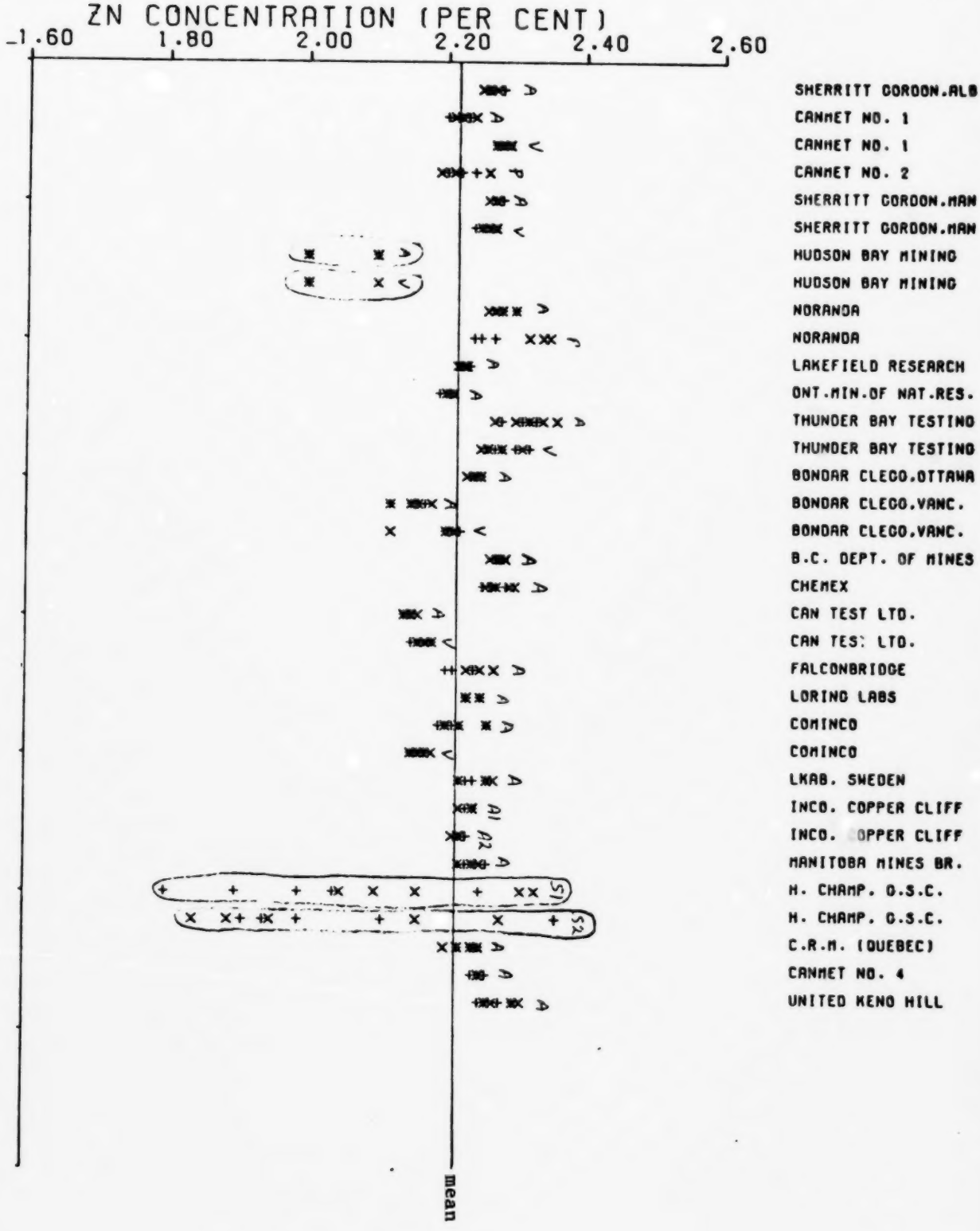


Figure 1(a) STANDARD REFERENCE MATERIAL RU-1, ZINC

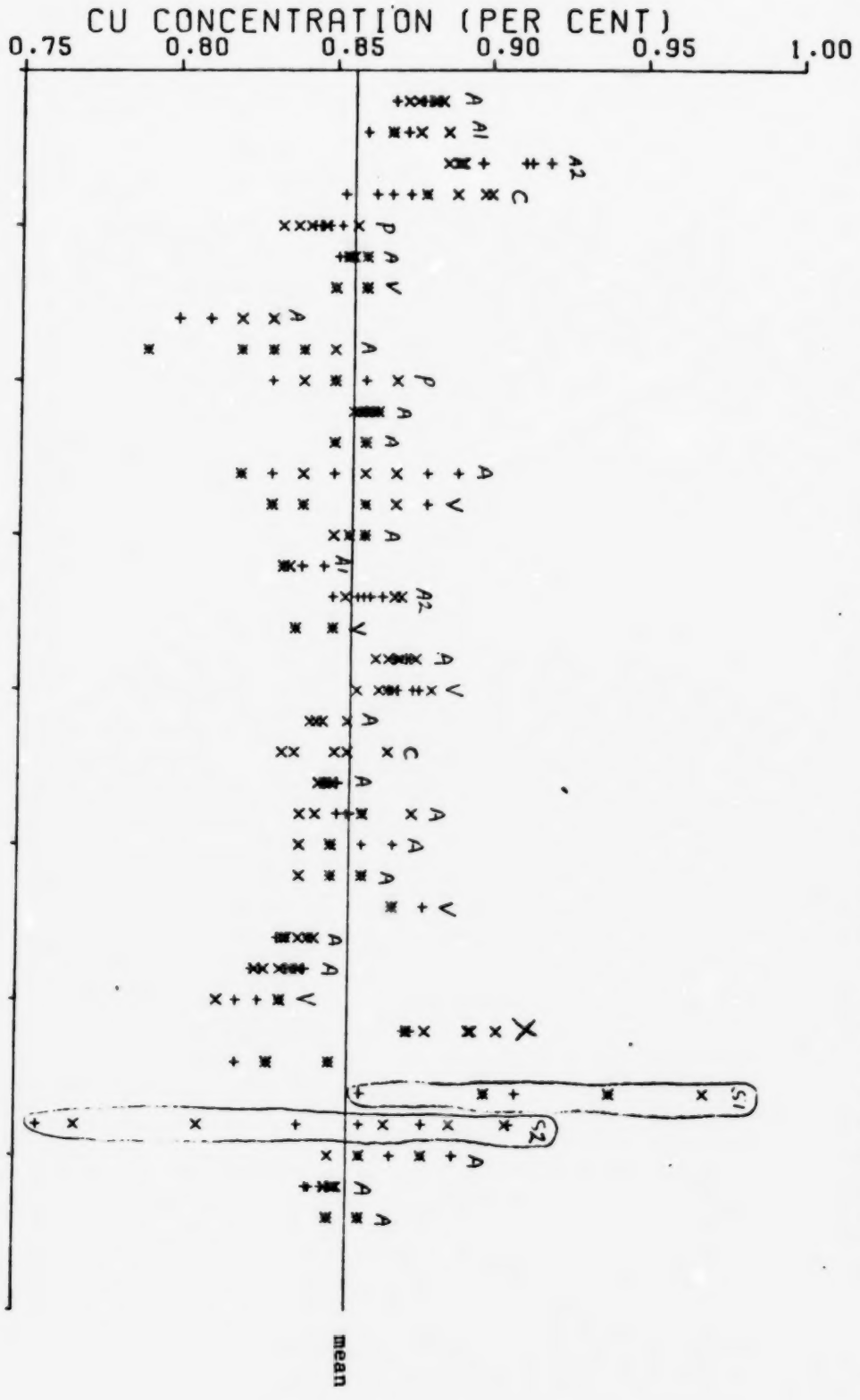


Figure 1(b) STANDARD REFERENCE MATERIAL RU-1, COPPER

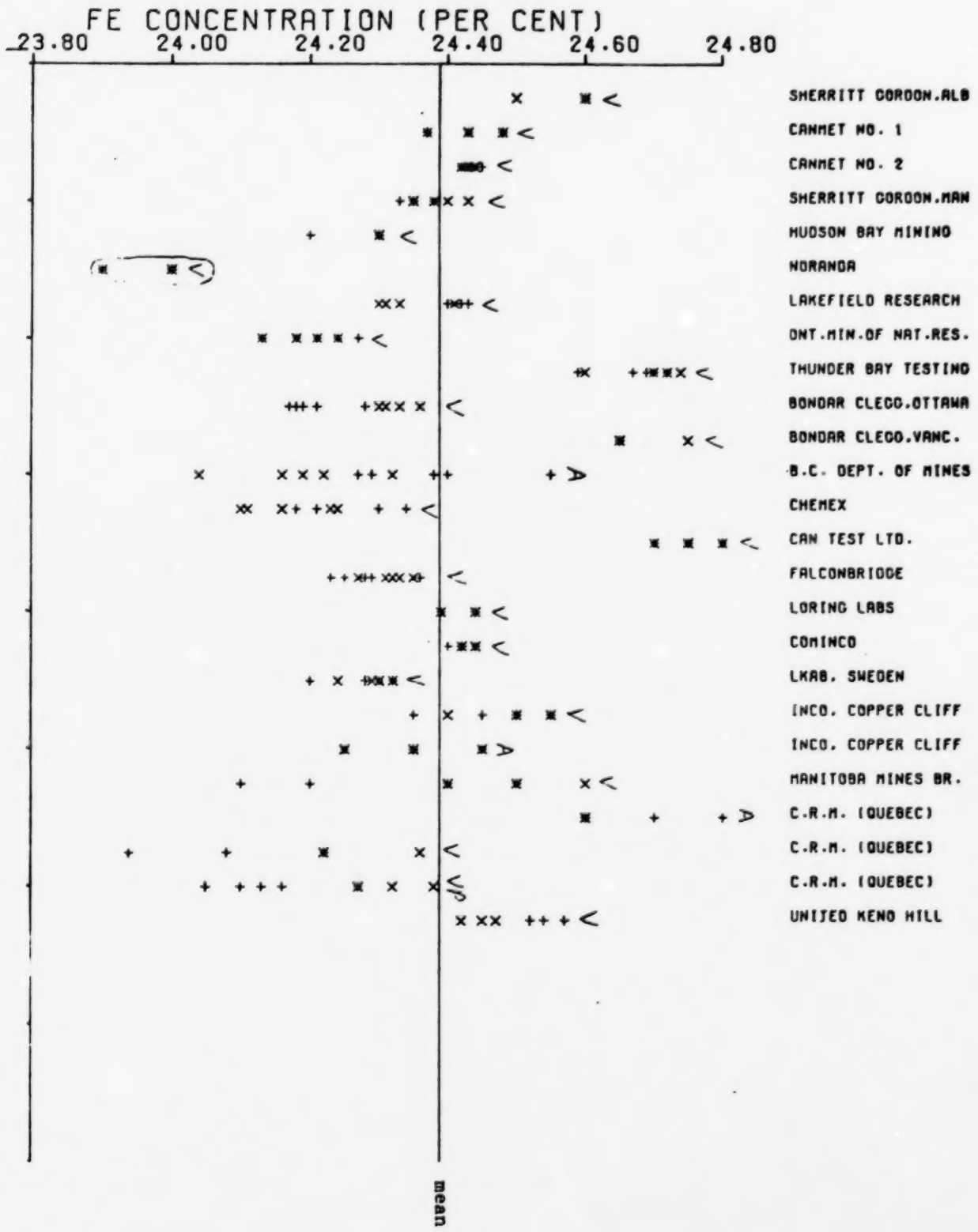


Figure 1(c) STANDARD REFERENCE MATERIAL RU-1, IRON

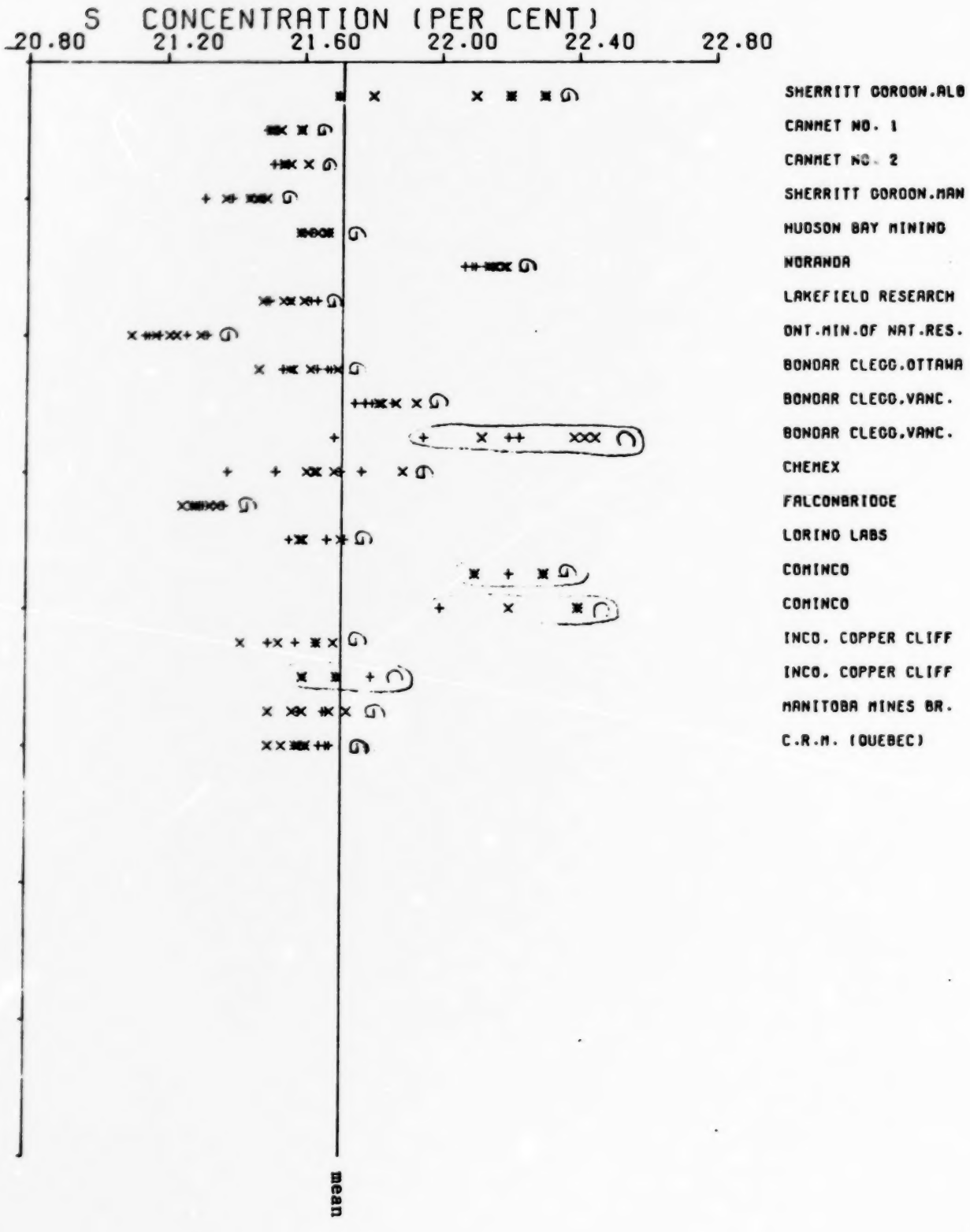


Figure 1(d) STANDARD REFERENCE MATERIAL RU-1, SULPHUR

Table 2(a)

Laboratory means, coefficients of variation, and summary of t-tests on between-bottle zinc results for RU-1

		BOTTLE 1			BOTTLE 2			NULL HYPOTH.	OVERALL			
		N	MEAN	ST.DEV.	N	MEAN	ST.DEV.		N	MEAN	ST.DEV.	C.V.(%)
LAB- 1	(A.A.)	5	2.2580	.0084	5	2.2720	.0110	A	10	2.2650	.0118	.52
LAB- 2	(A.A.)	5	2.2220	.0110	5	2.2220	.0130	A	10	2.2220	.0114	.51
LAB- 2	(VOL.)	5	2.2940	.0089	5	2.2780	.0084	A	10	2.2810	.0088	.38
LAB- 3	(POLAR.)	5	2.2120	.0286	5	2.2240	.0167	A	10	2.2180	.0230	1.04
LAB- 4	(A.A.)	5	2.2680	.0045	5	2.2740	.0055	A	10	2.2710	.0057	.25
LAB- 4	(VOL.)	5	2.2640	.0089	5	2.2540	.0114	A	10	2.2590	.0110	.49
*LAB- 5	(A.A.)	5	2.0600	.0548	5	2.0400	.0548	A	10	2.0500	.0527	2.57
*LAB- 5	(VOL.)	5	2.0200	.0447	5	2.0000	0.0000	A	10	2.0100	.0316	1.57
LAB- 6	(A.A.)	5	2.2780	.0148	5	2.2820	.0110	A	10	2.2800	.0125	.55
LAB- 6	(POLAR.)	5	2.3340	.0134	5	2.2540	.0152	REJECT	10	2.2940	.0443	1.93
LAB- 7	(A.A.)	5	2.2240	.0060	5	2.2272	.0059	A	10	2.2256	.0059	.26
LAB- 8	(A.A.)	5	2.2060	.0055	5	2.2000	.0071	A	10	2.2030	.0067	.31
LAB-10	(A.A.)	5	2.3180	.0349	5	2.3140	.0207	A	10	2.3160	.0272	1.17
LAB-10	(VOL.)	5	2.2720	.0239	5	2.2860	.0241	A	10	2.2790	.0238	1.04
LAB-11	(A.A.)	5	2.2400	.0100	5	2.2460	.0055	A	10	2.2430	.0082	.37
LAB-12	(A.A.)	5	2.1520	.0217	5	2.1520	.0192	A	10	2.1520	.0193	.90
LAB-12	(VOL.)	5	2.1880	.0383	5	2.2100	.0100	A	10	2.1990	.0288	1.31
LAB-13	(A.A.)	5	2.2780	.0095	5	2.2770	.0043	A	10	2.2775	.0069	.31
LAB-14	(A.A.)	5	2.2780	.0180	5	2.2690	.0121	A	10	2.2735	.0152	.67
LAB-15	(A.A.)	5	2.1480	.0084	5	2.1460	.0055	A	10	2.1470	.0067	.31
LAB-15	(VOL.)	5	2.1680	.0084	5	2.1620	.0130	A	10	2.1650	.0108	.50
LAB-16	(A.A.)	5	2.2500	.0200	5	2.2200	.0187	REJECT	10	2.2350	.0242	1.08
LAB-18	(A.A.)	5	2.2340	.0089	5	2.2340	.0089	A	10	2.2340	.0084	.38
LAB-19	(A.A.)	5	2.2160	.0261	5	2.2160	.0270	A	10	2.2160	.0250	1.13
LAB-19	(VOL.)	5	2.1620	.0130	5	2.1640	.0089	A	10	2.1630	.0106	.49
LAB-20	(A.A.)	5	2.2540	.0195	5	2.2420	.0179	A	10	2.2480	.0187	.83
LAB-22	(A.A.-1)	5	2.2360	.0089	5	2.2380	.0045	A	10	2.2370	.0067	.30
LAB-22	(A.A.-2)	5	2.2180	.0045	5	2.2260	.0055	REJECT	10	2.2220	.0063	.28
LAB-25	(A.A.)	5	2.2400	.0122	5	2.2360	.0152	A	10	2.2380	.0132	.59
*LAB-26	(SPECTR.1)	5	2.1900	.1251	5	1.9960	.1669	A	10	2.0930	.1735	8.29
*LAB-26	(SPECTR.2)	5	2.0240	.1880	5	2.0620	.1832	A	10	2.0430	.1761	8.62
LAB-27	(A.A.)	5	2.2220	.0228	5	2.2340	.0134	A	10	2.2280	.0187	.84
LAB-28	(A.A.)	5	2.2500	0.0000	5	2.2500	.0071	A	10	2.2500	.0047	.21
LAB-29	(A.A.)	5	2.2800	.0235	5	2.2700	.0200	A	10	2.2750	.0212	.93
								TOTAL	340	2.2151	.0854	3.86

*Sets judged to be outliers.

Table 2(b)

Laboratory means, coefficients of variation, and summary of t-tests on between-bottle copper results for RU-1

		BOTTLE 1			BOTTLE 2			NULL HYPOTH.	OVERALL			
		N	MEAN	ST.DEV.	N	MEAN	ST.DEV.		N	MEAN	ST.DEV.	C.V.(%)
LAB- 1	(A.A.)	5	.8792	.0045	5	.8782	.0054	A	10	.8787	.0048	.54
LAB- 2	(A.A.-1)	5	.8752	.0075	5	.8684	.0053	A	10	.8718	.0071	.82
LAB- 2	(A.A.-2)	5	.8892	.0019	5	.9074	.0099	REJECT	10	.8983	.0117	1.31
LAB- 2	(COLOR.)	5	.8890	.0100	5	.8674	.0101	REJECT	10	.8782	.0148	1.69
LAB- 3	(POLAR.)	5	.8434	.0092	5	.8468	.0034	A	10	.8451	.0068	.80
LAB- 4	(A.A.)	5	.8562	.0023	5	.8548	.0033	A	10	.8555	.0028	.33
LAB- 4	(VOL.)	5	.8540	.0055	5	.8580	.0045	A	10	.8560	.0052	.60
LAB- 5	(A.A.)	5	.8240	.0055	5	.8040	.0055	REJECT	10	.8140	.0117	1.44
LAB- 6	(A.A.)	5	.8260	.0230	5	.8220	.0192	A	10	.8240	.0201	2.44
LAB- 6	(POLAR.)	5	.8520	.0110	5	.8480	.0110	A	10	.8500	.0105	1.24
LAB- 7	(A.A.)	5	.8600	.0032	5	.8608	.0023	A	10	.8604	.0026	.31
LAB- 8	(A.A.)	5	.8540	.0055	5	.8580	.0045	A	10	.8560	.0052	.60
LAB-10	(A.A.)	5	.8460	.0195	5	.8540	.0305	A	10	.8500	.0245	2.88
LAB-10	(VOL.)	5	.8460	.0182	5	.8480	.0217	A	10	.8470	.0189	2.23
LAB-11	(A.A.)	5	.8540	.0042	5	.8590	.0022	REJECT	10	.8565	.0041	.48
LAB-12	(A.A.-1)	5	.8344	.0009	5	.8390	.0054	A	10	.8367	.0044	.52
LAB-12	(A.A.-2)	5	.8672	.0074	5	.8592	.0059	A	10	.8632	.0076	.88
LAB-12	(VOL.)	5	.8428	.0066	5	.8428	.0066	A	10	.8428	.0062	.74
LAB-13	(A.A.)	5	.8704	.0048	5	.8726	.0024	A	10	.8715	.0038	.43
LAB-13	(VOL.)	5	.8686	.0088	5	.8722	.0046	A	10	.8704	.0069	.79
LAB-14	(A.A.)	THERE IS ONLY 1 BOTTLE							5	.8470	.0047	.55
LAB-14	(COLOR.)	THERE IS ONLY 1 BOTTLE							5	.8492	.0137	1.61
LAB-15	(A.A.)	5	.8476	.0017	5	.8500	.0020	A	10	.8488	.0021	.25
LAB-16	(A.A.)	5	.8562	.0142	5	.8558	.0029	A	10	.8560	.0097	1.13
LAB-18	(A.A.)	5	.8460	.0055	5	.8560	.0089	A	10	.8510	.0088	1.03
LAB-19	(A.A.)	5	.8500	.0100	5	.8560	.0055	A	10	.8530	.0082	.97
LAB-19	(VOL.)	5	.8700	.0000	5	.8720	.0045	A	10	.8710	.0032	.36
LAB-20	(A.A.)	5	.8398	.0043	5	.8366	.0038	A	10	.8382	.0042	.50
LAB-22	(A.A.)	5	.8334	.0059	5	.8338	.0071	A	10	.8336	.0061	.74
LAB-22	(VOL.)	5	.8300	.0089	5	.8298	.0063	A	10	.8299	.0073	.88
LAB-24	(X.R.F.)	5	.8902	.0119	5	.8752	.0008	REJECT	10	.8827	.0112	1.27
LAB-25	(A.A.)	5	.8340	.0089	5	.8320	.0110	A	10	.8330	.0095	1.14
* LAB-26	(SPECTR.1)	5	.9380	.0249	5	.8940	.0344	REJECT	10	.9160	.0366	3.99
* LAB-26	(SPECTR.2)	5	.8482	.0579	5	.8490	.0572	A	10	.8486	.0543	6.40
LAB-27	(A.A.)	5	.8660	.0134	5	.8780	.0130	A	10	.8720	.0140	1.60
LAB-28	(A.A.)	5	.8508	.0018	5	.8470	.0033	A	10	.8489	.0032	.38
LAB-29	(A.A.)	5	.8540	.0055	5	.8560	.0055	A	10	.8550	.0053	.62
								TOTAL	360	.8559	.0242	2.83

*Sets judged to be outliers.

Table 2(c)

Laboratory means, coefficients of variation, and summary of t-tests on between-bottle iron results for RU-1

		BOTTLE 1			BOTTLE 2			NULL HYPOTH.	OVERALL			
		N	MEAN	ST.DEV.	N	MEAN	ST.DEV.		N	MEAN	ST.DEV.	C.V.(%)
LAB- 1	(VOL.)	5	24.5800	.0447	5	24.6000	.0000	A	10	24.5900	.0316	.13
LAB- 2	(VOL.)	5	24.4280	.0390	5	24.4260	.0550	A	10	24.4270	.0450	.18
LAB- 3	(VOL.)	5	24.4340	.0089	5	24.4340	.0114	A	10	24.4340	.0097	.04
LAB- 4	(VOL.)	5	24.3820	.0342	5	24.3520	.0179	A	10	24.3670	.0302	.12
LAB- 5	(VOL.)	5	24.3000	.0000	5	24.2800	.0447	A	10	24.2900	.0316	.13
*LAB- 6	(VOL.)	5	23.9800	.0447	5	23.9800	.0447	A	10	23.9800	.0422	.18
LAB- 7	(VOL.)	5	24.3320	.0449	5	24.4160	.0152	REJECT	10	24.3740	.0544	.22
LAB- 8	(VOL.)	5	24.1880	.0409	5	24.2060	.0541	A	10	24.1970	.0462	.19
LAB-10	(VOL.)	5	24.7000	.0583	5	24.6740	.0503	A	10	24.6870	.0531	.22
LAB-11	(VOL.)	5	24.3200	.0255	5	24.2060	.0439	REJECT	10	24.2630	.0690	.28
LAB-12	(VOL.)	5	24.6900	.0548	5	24.6500	.0000	A	10	24.6700	.0422	.17
LAB-13	(A.A.)	5	24.1860	.1014	5	24.3780	.1112	REJECT	10	24.2820	.1425	.59
LAB-14	(VOL.)	5	24.1680	.0653	5	24.2660	.0677	REJECT	10	24.2170	.0812	.34
LAB-15	(VOL.)	5	24.7500	.0354	5	24.7500	.0354	A	10	24.7500	.0333	.13
LAB-16	(VOL.)	5	24.3160	.0297	5	24.2820	.0497	A	10	24.2990	.0425	.18
LAB-18	(VOL.)	5	24.4200	.0274	5	24.4300	.0224	A	10	24.4250	.0242	.10
LAB-19	(VOL.)	5	24.4320	.0110	5	24.4200	.0141	A	10	24.4260	.0135	.06
LAB-20	(VOL.)	5	24.2880	.0295	5	24.2760	.0456	A	10	24.2820	.0368	.15
LAB-22	(VOL.)	5	24.4800	.0758	5	24.4700	.0758	A	10	24.4750	.0717	.29
LAB-22	(A.A.)	5	24.3300	.0837	5	24.3500	.0707	A	10	24.3400	.0738	.30
LAB-25	(VOL.)	5	24.4800	.0837	5	24.2800	.1643	REJECT	10	24.3800	.1619	.66
LAB-27	(A.A.)	5	24.6000	.0000	5	24.6600	.0894	A	10	24.6300	.0675	.27
LAB-27	(VOL.)	5	24.2760	.0767	5	24.1080	.1171	REJECT	10	24.1920	.1287	.53
LAB-27	(VOL.-P.)	5	24.3020	.0487	5	24.1420	.0823	REJECT	10	24.2220	.1057	.44
LAB-29	(VOL.)	5	24.4520	.0205	5	24.5440	.0251	REJECT	10	24.4980	.0531	.22
								TOTAL	250	24.3879	.1896	.78

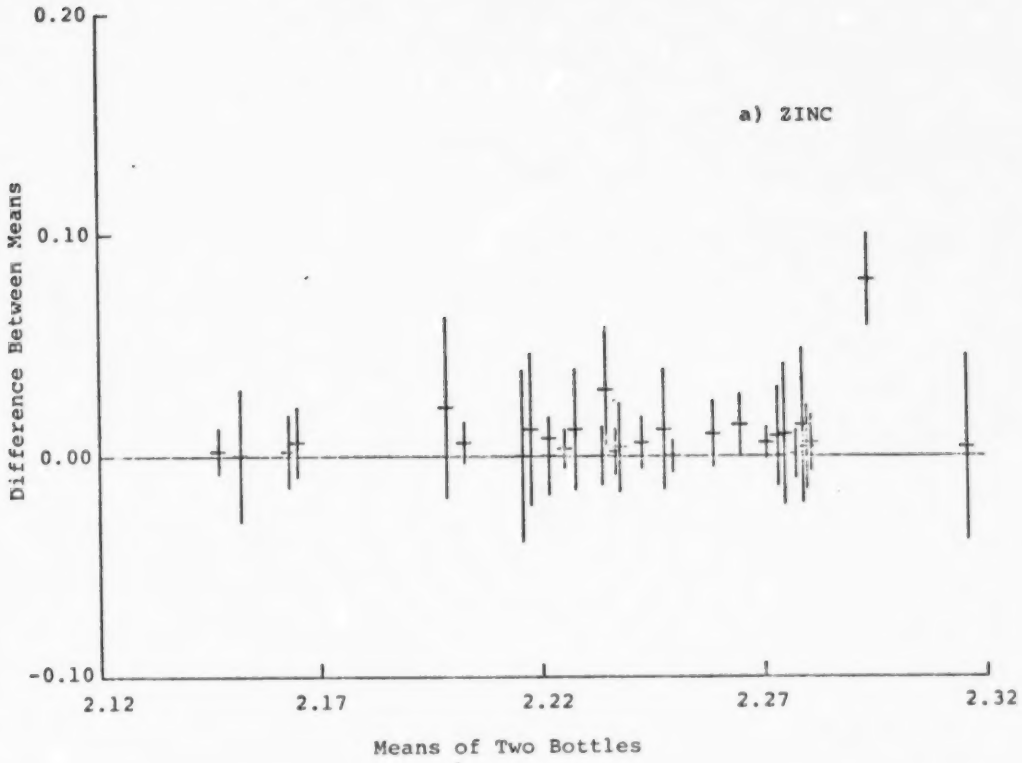
*Set judged to be outlier.

Table 2(d)

Laboratory means, coefficients of variation, and summary of t-tests on between-bottle sulphur results for RU-1

	BOTTLE 1			BOTTLE 2			NULL HYPOTH.	OVERALL			
	N	MEAN	ST.DEV.	N	MEAN	ST.DEV.		N	MEAN	ST.DEV.	C.V.(%)
LAB- 1 (GRAV.)	5	22.0200	.2588	5	22.0200	.2950	A	10	22.0200	.2616	1.19
LAB- 2 (GRAV.)	5	21.5440	.0434	5	21.5200	.0400	A	10	21.5320	.0413	.19
LAB- 3 (GRAV.)	5	21.5580	.0303	5	21.5280	.0179	A	10	21.5430	.0293	.13
LAB- 4 (GRAV.)	5	21.4460	.0462	5	21.4180	.0698	A	10	21.4320	.0577	.27
LAB- 5 (GRAV.)	5	21.6340	.0336	5	21.6440	.0371	A	10	21.6390	.0338	.16
LAB- 6 (GRAV.)	5	22.1640	.0207	5	22.0920	.0249	REJECT	10	22.1280	.0437	.20
LAB- 7 (GRAV.)	5	21.5560	.0498	5	21.5620	.0680	A	10	21.5590	.0563	.26
LAB- 8 (GRAV.)	5	21.2020	.0740	5	21.2100	.0775	A	10	21.2060	.0715	.34
LAB-11 (GRAV.)	5	21.5840	.0844	5	21.6140	.0691	A	10	21.5990	.0745	.34
LAB-12 (GRAV.)	5	21.8540	.0472	5	21.7940	.0410	A	10	21.8240	.0523	.24
* LAB-12 (COMB.)	5	22.3660	.1397	5	22.0600	.2379	REJECT	10	22.2130	.2446	1.10
LAB-14 (GRAV.)	5	21.6920	.1145	5	21.5800	.1583	A	10	21.6360	.1430	.66
LAB-16 (GRAV.)	5	21.3000	.0400	5	21.3100	.0354	A	10	21.3050	.0360	.17
LAB-18 (GRAV.)	5	21.6180	.0517	5	21.6580	.0589	A	10	21.6380	.0563	.26
* LAB-19 (GRAV.)	5	22.2200	.1095	5	22.2200	.0837	A	10	22.2200	.0919	.41
* LAB-19 (COMB.)	5	22.3600	.0894	5	22.2400	.2191	A	10	22.3000	.1700	.76
LAB-22 (GRAV.)	5	21.5840	.1088	5	21.5720	.0701	A	10	21.5780	.0865	.40
* LAB-22 (COMB.)	5	21.6400	.0548	5	21.7000	.0707	A	10	21.6700	.0675	.31
LAB-25 (GRAV.)	5	21.6160	.0907	5	21.6200	.0412	A	10	21.6180	.0665	.31
LAB-27 (GRAV.)	5	21.5640	.0439	5	21.6380	.0421	REJECT	10	21.6010	.0563	.26
							TOTAL	200	21.7130	.3172	1.46

*Sets judged to be outliers.



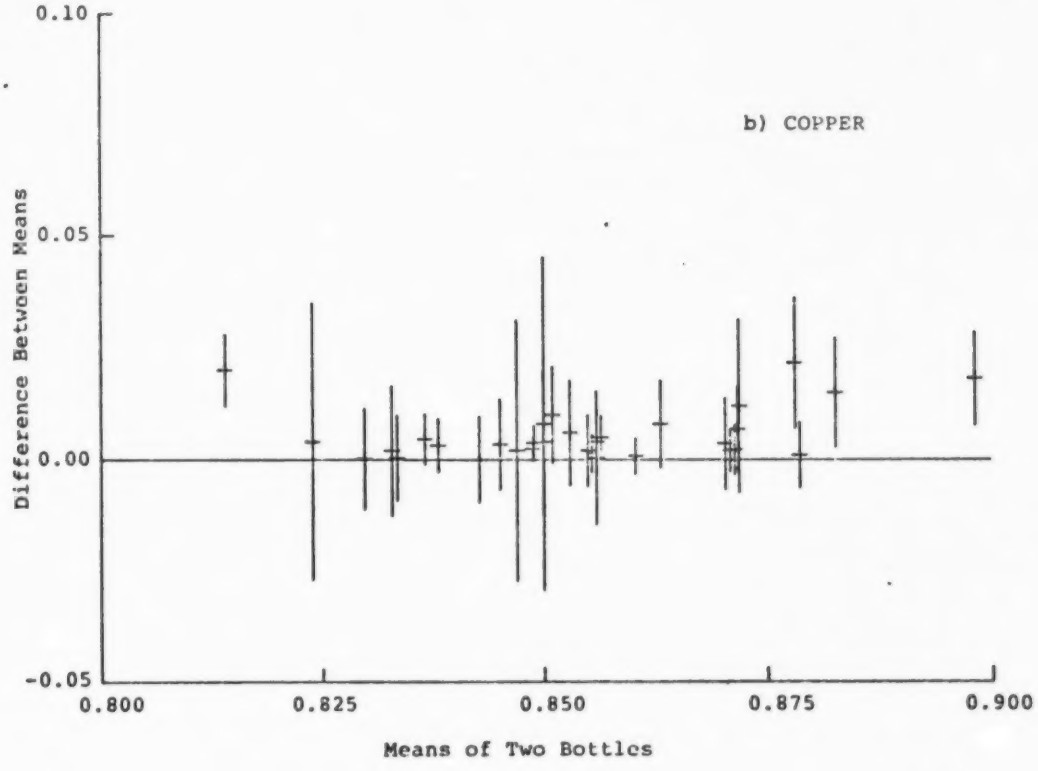
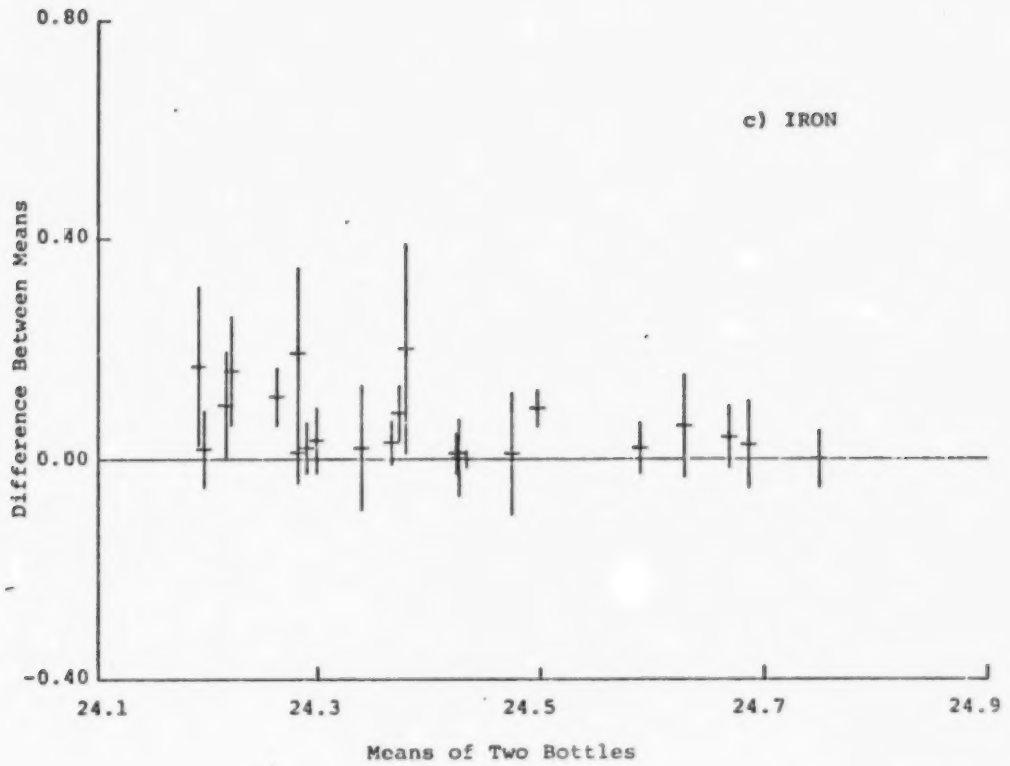


Figure 2. Illustration of degree of homogeneity



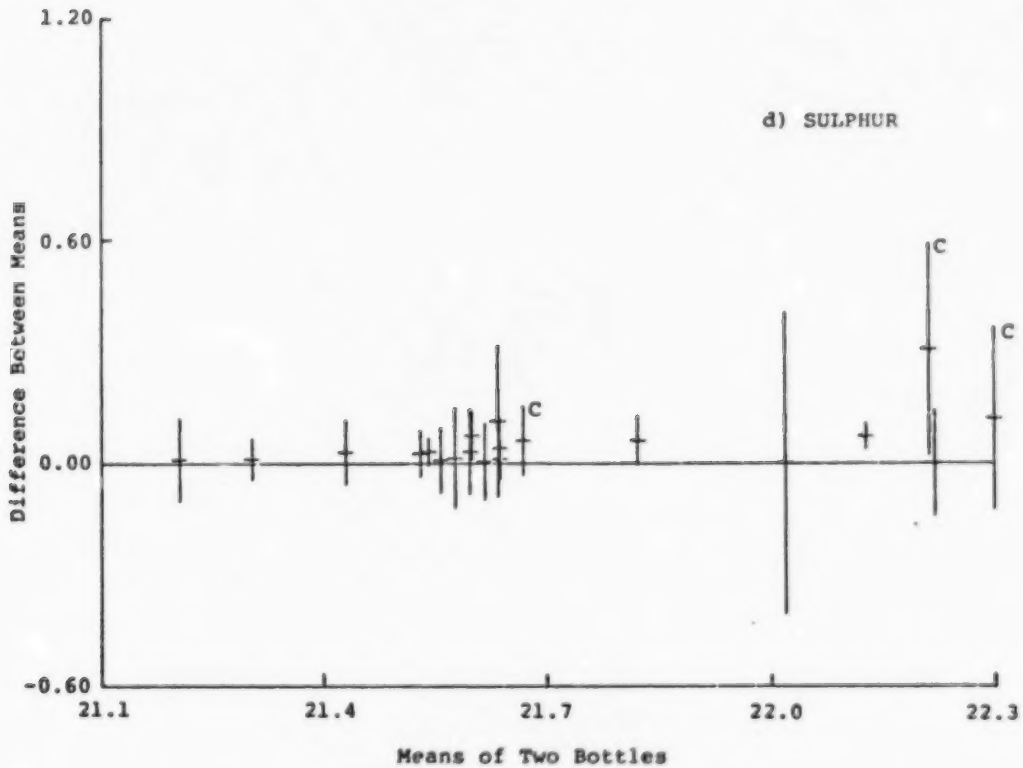


Figure 2 (continued)

Table 3(a)

STANDARD REFERENCE MATERIAL RU-1. ZINC

ANALYSIS OF VARIANCE TABLE

SOURCE OF VARIATION	D.F.	SUM OF SQUARES	MEAN SQUARE	F-RATIO	F-TABLE *
BETWEEN SETS	29	.52266008E+00	.18022761E-01	23.497	1.847
BETWEEN BOTTLES	30	.23011600E-01	.76702000E-03	3.141	1.507
BETWEEN REPLICATES	240	.58600800E-01	.24417000E-03		
TOTAL	299	.60427148E+00			

PSI	=	.41540031E-01
OMEGA	=	.10225947E-01
SIGMA	=	.15625940E-01
GRAND MEAN	=	3.2372

* 95% probability

Table 3(b)

STANDARD REFERENCE MATERIAL BU-1. COPPER

ANALYSIS OF VARIANCE TABLE

SOURCE OF VARIATION	D.F.	SUM OF SQUARES	MEAN SQUARE	F-RATIO	F-TABLE*
BETWEEN SETS	32	.10460695E+00	.32689672E-02	20.876	1.793
BETWEEN BOTTLES	33	.51674000E-02	.15658788E-03	1.778	1.481
BETWEEN REPLICATES	264	.23254000E-01	.88083333E-04		
TOTAL	329	.13302835E+00			

RSI	=	.17641337E-01
OMEGA	=	.37014734E-02
SIGMA	=	.93852722E-02
GRAND MEAN	=	.2345

* 95% probability

Table 3(c)

STANDARD REFERENCE MATERIAL RU-1. IRON

ANALYSIS OF VARIANCE TABLE

SOURCE OF VARIATION	D.F.	SUM OF SQUARES	MEAN SQUARE	F-RATIO	F-TABLE*	
BETWEEN SETS	23	.60944263E+01	.26475766E+00	14.219	1.993	
BETWEEN BOTTLES	24	.44683000E+00	.18620417E-01	5.392	1.574	1 23 1
BETWEEN REPLICATES	142	.48302000E+00	.34535417E-02			
TOTAL	239	.71999962E+01				

PSI = .15442762E+00

OMEGA = .55976084E-01

SIGMA = .58766842E-01

GRAND MEAN = 24.4046

* 95% probability

Table 3(d)

STANDARD REFERENCE MATERIAL RU-1. SULPHUR

ANALYSIS OF VARIANCE TABLE

SOURCE OF VARIATION	D.F.	SUM OF SQUARES	MEAN SQUARE	F-RATIO	F-TABLE*
BETWEEN SETS	15	.78630375E+01	.52420650E+00	104.763	2.352
BETWEEN BOTTLES	16	.80060000E-01	.5003750UE-02	.569	1.723
BETWEEN REPLICATES	128	.11243400E+01	.87878125E-02		
TOTAL	159	.30679375E+01			

PSI = .22746021E+00

OMEGA = 0.

SIGMA = .93743333E-01

GRAND MEAN = 21.6161

* 95% probability

TABLE 4

Estimation of Statistical Parameters for RU-1 (After Rejection of Outliers)

Element	No. of Labs	No. of Sets	Total No. of Results	Median	Mean	95% Conf. Interval		Spread %	Average Within-Set cv, %	Cert. Factor	Within-Set Variance	Between-Set Variance	Variance of the Grand Mean	Skewness	Kurtosis
						Low	High								
Zinc	24	30	300	2.240	2.237	2.221	2.253	1.42	0.66	2.14	0.3023×10^{-3}	0.1772×10^{-2}	0.6008×10^{-4}	-0.30	3.08
Copper	22	35	340	0.853	0.854	0.848	0.861	1.44	0.97	1.48	0.9592×10^{-4}	0.3082×10^{-3}	0.9214×10^{-5}	0.01	3.65
Iron	21	24	240	24.40	24.40	24.34	24.47	0.56	0.25	2.27	0.5139×10^{-2}	0.2596×10^{-1}	0.1103×10^{-2}	0.28	2.60
Sulphur	16	16	160	21.59	21.62	21.49	21.74	1.13	0.34	3.36	0.8367×10^{-2}	0.5158×10^{-1}	0.3276×10^{-2}	0.77	3.98

POOR

QUALITY

ORIGINAL